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(54) Title: FOODS CONTAINING THERMALLY-INHIBITED STARCHES AND FLOURS

## (57) Abstract

A granular starch or flour which is both thermally-inhibited and pregelatinized is used as an ingredient in various foods. The starches are functionally equivalent to chemically cross-linked starches. The starches or flours are prepared by heat-treating the starch to substantially anhydrous, preferably at a neutral or basic pH, for a time sufficient to inhibit the starch to the desired degree. The starch may be pregelatinized prior to or after thermal inhibition using known methods which do not substantially rupture the starch granules.

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FOODS CONTAINING THERMALLY-INHIBITED  
STARCHES AND FLOURS

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Background Of The Invention

This invention relates to foods containing thermally-inhibited starches and flours which are functionally equivalent to chemically inhibited (i.e., crosslinked) starches.

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The processed food industry has sought to satisfy consumer demands for foods containing starches which have not been chemically modified but which have the same functional properties as chemically modified starches. Starches are chemically modified with difunctional reagents, such as phosphorus oxychloride, sodium trimetaphosphate, adipic and acetic anhydrides and epichlorohydrin, to produce chemically crosslinked starches having excellent tolerance to processing variables such as heat, shear, and pH extremes. Such chemically crosslinked starches provide a desirable smooth texture and possess viscosity stability throughout

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the processing operation and normal shelf life of the food. In contrast, unmodified starches breakdown in viscosity, loose thickening capacity and textural qualities, and behave unpredictably during storage as a result of the stresses encountered during food processing. Heat, shear, and/or an extreme pH, especially an acidic pH, tend to fully disrupt the starch granules and disperse the starch polymers into the food. Hence, unmodified starches are generally unsuitable for use in processed foods.

#### Summary Of The Invention

The present invention provides a food which contains a thermally-inhibited starch or flour which is a granular pregelatinized starch or flour. When the starch is pregelatinized prior to thermal inhibition, the starch is referred to as a "pregelatinized thermally-inhibited granular starch". When the starch is pregelatinized subsequent to thermal inhibition, the starch is referred to as a "thermally-inhibited pregelatinized granular starch."

The thermally-inhibited starches can be pregelatinized prior to or after the thermal inhibition process using methods known in the art which will not destroy the granular structure. The resulting pregelatinized starches are particularly useful in food applications where cold-water-soluble or instant gelling starches are used.

The starches or flours are thermally-inhibited in a process that results in the starch or flour becoming and remaining inhibited, hereinafter referred to as inhibited or thermally-inhibited, without the addition of chemical reagents. When these thermally-inhibited starches or flours are dispersed and/or cooked in water, they exhibit the properties characteristic of an

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inhibited pregelatinized starch. The amount of thermal inhibition required will depend on the reason the starch was included in the food, e.g., thickening, gelling, extending, and the like, as well as the particular processing conditions used to prepare the foods. Foods prepared with the thermally-inhibited and pregelatinized starches or flours possess both process tolerance, such as resistance to heat, acid, and shear, as well as improved texture and viscosity stability. These thermally-inhibited starches have the functional characteristics of a chemically crosslinked starch.

Initially, it should be noted that when certain native starches, particularly waxy-based starches, are gelatinized, they reach a peak viscosity, which soon begins to breakdown as the starch polymers disassociate and become solubilized and the granular integrity is lost, thereby causing the starch paste to become cohesive or runny. In contrast, when the thermally-inhibited and pregelatinized starches are dispersed and/or cooked in an aqueous medium, the starch granules are more resistant to viscosity breakdown than starches which are not thermally-inhibited. This resistance to breakdown results in what is subjectively considered a non-cohesive, or "short" textured paste, meaning that the gelatinized starch tends to be salve-like and heavy in viscosity rather than runny or gummy.

Depending on the extent of the heat treatment, various levels of inhibition can be achieved. For example, higher viscosity products with little breakdown as well as highly inhibited, low viscosity products with no breakdown can be prepared by the thermal inhibition process described herein.

The present invention also provides a method for preparing a food which is to be thickened or gelled, with or without heating, which comprises the step of

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adding to one or more of the food ingredients, prior to  
or during the processing of the food ingredients, a  
thermally-inhibited and pregelatinized granular starch or  
flour. It also provides a method for extending a food  
5 which comprises the step of replacing or partially  
replacing one or more of the ingredients typically used  
in the food (e.g., fat, fruit solids, and the like) with  
a thermally-inhibited and pregelatinized starch. One  
skilled in the art will recognize that mixtures of  
10 various starches can be used.

Foods containing the thermally-inhibited and  
pregelatinized starches or flours are functionally  
equivalent to foods containing chemically crosslinked  
starches. This permits the food manufacturer to avoid  
15 label declarations associated with the chemical  
crosslinking of starch, and, thereby, provide a perceived  
consumer benefit.

Description Of The Preferred Embodiments

20 The thermally inhibited starches and flours of  
this invention can be derived from any native source.  
Typical sources for the starches and flours are cereals,  
tubers, roots, legumes, and fruits. The native source  
can be corn, pea, potato, sweet potato, banana, barley,  
25 wheat, rice, sago, amaranth, tapioca, sorghum, waxy  
maize, waxy rice, waxy barley, waxy potato, waxy sorghum,  
starches containing greater than 40% amylose (also  
referred to as high amylose starches), and the like.  
Unless specifically distinguished, references to starch  
30 is meant to include the corresponding flour. References  
to starch are also meant to include starch to which  
protein has been added, whether the protein is endogenous  
protein, or added protein from an animal or plant source,  
such as, zein, albumin, and soy protein.

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As used herein, a native starch is one as it is found in nature in unmodified form.

The first step of the thermal inhibition process is dehydrating the starch for a time and at a temperature sufficient to render the starch anhydrous or substantially anhydrous. The second step is heat treating the anhydrous or substantially anhydrous starch for a time and at a temperature sufficient to inhibit the starch. As used herein, substantially anhydrous means less than 1% moisture by weight.

Native granular starches have a natural pH of about 5.0 to 6.5. When such starches are heated to temperatures above about 125°C in the presence of water acid hydrolysis, i.e., degradation, of the starch occurs.

This degradation impedes or prevents inhibition. Therefore, the dehydration conditions need to be chosen so that degradation is avoided. Suitable conditions are dehydrating at low temperatures and the starch's natural pH or dehydrating at higher temperatures after increasing the pH of the starch to neutral or above. The preferred pH ranges are a pH of at least 7.0, preferably pH 7.5 to pH 10.5. The most preferred pH ranges are above pH 8 to below pH 10. At a pH above 12, gelatinization more easily occurs. Therefore, pH adjustments below 12 are more effective. It should be noted that the textural and viscosity benefits of the thermal inhibition process tend to be enhanced as the pH is increased, although higher pHs tend to increase browning of the starch during the heat treating step.

If the pregelatinization process is performed first, a granular starch or flour is slurried in water in a ratio of 1.5 to 2.0 parts to 1.0 part starch, and optionally, the pH is adjusted to neutral or greater. As used herein, neutral covers the range of pH values around pH 7 and is meant to include from about pH 6.5 to about

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pH 7.5. The slurry is simultaneously pregelatinized and dried and the dried starch or flour is thermally inhibited. The thermal inhibition process comprises the steps of dehydrating the pregelatinized starch until it  
5 is anhydrous or substantially anhydrous, which for purposes herein means containing less than 1% moisture by weight, and then heat treating the anhydrous or substantially anhydrous starch or flour at a temperature and for a period of time effective to cause inhibition.

10 Alternatively, if the thermal inhibition process is performed first, the starch or flour is slurried in water; optionally, the pH of the starch or flour is adjusted to neutral or greater; and the starch or flour is dried to about 2.15% moisture. The starch or  
15 flour is then dried to anhydrous or substantially anhydrous, and heat treated at a temperature and for a period of time effective to cause inhibition. The inhibited starch or flour is reslurried in water, optionally pH adjusted, and simultaneously pregelatinized  
20 and dried.

Suitable bases for use in the pH adjustment step include, but are not limited to, sodium hydroxide, sodium carbonate, tetrasodium pyrophosphate, ammonium orthophosphate, disodium orthophosphate, trisodium  
25 phosphate, calcium carbonate, calcium hydroxide, potassium carbonate, and potassium hydroxide, and any other bases approved for use under Food and Drug Administration laws or other regulatory laws. The preferred base is sodium carbonate. It may be possible  
30 to use bases not approved under the above regulations provided they can be washed from the starch so that the final product conforms to good manufacturing practices for pharmaceutical use.

After the pH adjustment, the starch slurry is  
35 then either dewatered and dried, or dried directly,

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typically to a 2-15% moisture content. These drying procedures are to be distinguished from the thermal inhibition process steps in which the starch is dehydrated to anhydrous or substantially anhydrous and  
5 heat treated.

Preferably, the temperatures used to dehydrate the starch are 125°C or less, more preferably 100° to 120°C. The dehydrating temperature can be lower than 100°C, but a temperature of at least 100°C will be more  
10 efficient for removing moisture.

After it is dehydrated, the starch is heat treated for a time and at a temperature sufficient to inhibit the starch. The preferred heating temperatures are greater than about 100°C. For practical purposes,  
15 the upper limit of the heat treating temperature is about 200°C. Typical temperatures are 120-180°C, preferably 140-160°C, most preferably 160°C. The temperature selected will depend upon the amount of inhibition desired and the rate at which it is to be achieved.  
20

The time at the final heating temperature will depend upon the level of inhibition desired. When a fluidized bed is used, the times range from 0 minutes to 20 hours, typically 0.5-3.0 hours. Longer times are required at lower temperatures to obtain more inhibited  
25 starches.

For most applications, the dehydrating and heat treating steps will be continuous and accomplished by the application of heat to the starch beginning from ambient temperature. The moisture will be driven off during the  
30 heating and the starch will become anhydrous or substantially anhydrous. Some level of inhibition will be attained simultaneously with the dehydration, even before the final heat treating temperature is reached. Usually, at these initial levels of inhibition, the peak  
35 viscosities are higher than the peak viscosities of

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starches heated for longer times, although there will be greater breakdown in viscosity from the peak viscosity. With continued heat treating, the peak viscosities are lower, but the viscosity breakdowns are less.

5 The process may be carried out as part of a continuous process involving the extraction of the starch from a plant material.

10 As will be seen in the following examples, the source of the starch, initial pH and moisture content of the starch, the dehydrating temperature, the heating time and temperature, and equipment used are all interrelated variables that affect the amount of inhibition.

15 The process steps may be performed at normal pressures, under vacuum or under pressure, and may be accomplished by conventional means known in the art. The preferred method is by the application of dry heat in air or in an inert gaseous environment.

20 The heat treating step can be carried out in the same apparatus in which the dehydration occurs, and most conveniently the process is continuous with the dehydration and heat treating occurring in the same apparatus, as when a fluidized bed reactor is used.

25 The dehydrating and heat treating apparatus can be any industrial ovens, conventional ovens, microwave ovens, dextrinizers, dryers, mixers and blenders equipped with heating devices and other types of heaters, provided that the apparatus is fitted with a vent to the atmosphere so that moisture does not accumulate and precipitate onto the starch. The preferred apparatus is 30 a fluidized bed. Preferably, the apparatus is equipped with a means for removing water vapor, such as, a vacuum or a blower to sweep air or the fluidizing gas from the head-space of the fluidized bed. Suitable fluidizing gases are air and nitrogen. For safety reasons, it is 35 preferable to use a gas containing less than 12% oxygen.

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5 Superior inhibited starches having high viscosities with low percentage breakdown in viscosity are obtained in shorter times in the fluidized bed reactor than can be achieved using other conventional heating ovens or dryers.

10 These starches and flours may be pregelatinized in such a way that a majority of the starch granules are swollen, but remain intact. The granules may be pregelatinized in this manner either prior to or after the thermal inhibition process. The resulting starches are pregelatinized thermally-inhibited granular starches or thermally-inhibited pregelatinized granular starches. Exemplary processes for preparing pregelatinized granular starches are disclosed in U.S. 4,280,851, U.S. 4,465,702, 15 U.S. 5,037,929, and U.S. 5,149,799.

15 The thermal inhibition process or pregelatinization process may be carried out prior to or after other starch reactions used to modify starch, such as, heat- and/or acid-conversion, oxidation, 20 phosphorylation, etherification, esterification, chemical crosslinking, enzyme modification, and the like. Usually these modifications are performed before the starch is thermally inhibited. Procedures for modifying starches are described in the chapter "Starch and Its 25 Modification" by M.W. Rutenberg, pages 22-26 to 22-47, Handbook of Water Soluble Gums and Resins, R.L. Davidson, Editor (McGraw-Hill, Inc., New York, NY 1980).

30 The starches may be inhibited individually or more than one may be inhibited at the same time. The starches may be inhibited in the presence of other materials or ingredients that would not interfere with the thermal inhibition process or alter the properties of the starch product.

35 Following the thermal inhibition step, the resulting starches may be screened to the desired

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particle size. The starch is a granular pregelatinized starch the starch can be washed by any known methods that will maintain granular integrity. If desired, the pH may be adjusted.

5 The thermally-inhibited and pregelatinized granular starches or flours, both non-pregelatinized, may be blended with other unmodified or modified starches, including pregelatinized starches, or with other food ingredients before use in a food product.

10 The starches and flours can be pregelatinized prior to or subsequent to the thermal inhibition process. The starches can be pregelatinized according to any of the known pregelatinization processes that will result in a majority of intact granules, which processes will be discussed hereafter. The amount of pregelatinization, and consequently, whether the starch will display a high or a low initial viscosity when dispersed in water, can be regulated by the pregelatinization procedures. In general, if the pregelatinization step is accomplished by spray-drying, the longer the residence time in the spray nozzle and the higher the ratio of steam to starch, the higher the initial viscosity of the pregelatinized granular starch when it is subsequently dispersed in water. Conversely, the lower the residence time and the lower the amount of heat and moisture, the lower the initial viscosity of the pregelatinized granular starch.

15 20 25 30 The starches can be pregelatinized according to any of the known pregelatinization processes that result in the maintenance of intact granules. Exemplary processes are disclosed in U.S. 4,280,851, U.S. 4,465,702, 5,037,929, and U.S. 5,149,799.

35 U.S. 4,280,851 (issued July 28, 1981 to Pitchen et al.) describes a dual-atomization spray-drying process for preparing granular pregelatinized starches. In this process a mixture of the granular starch in an aqueous

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solvent is injected through an atomization aperture in a nozzle assembly to form a finely divided spray. A heating medium is injected through a second aperture in the nozzle assembly into the spray of atomized starch to heat the starch to its gelatinization temperature. An enclosed chamber surrounds the atomization and heating medium injection apertures and defines a vent aperture positioned to enable the heated spray of starch to exit the chamber. The arrangement is such that the lapsed time between passage of the spray of the starch through the chamber from the atomization aperture to the vent aperture defines the gelatinization time of the starch. The resulting spray-dried pregelatinized starch comprises uniformly gelatinized starch granules in the form of indented spheres, with a majority of the granules being whole and unbroken and swelling upon rehydration.

An apparatus for carrying out this dual-atomization process is disclosed in U.S. 4,600,472 (issued July 13, 1986 to Pitchon et al.). Nozzles suitable for use in the preparation of these starches are described in U.S. 4,610,760 (issued September 9, 1986 to Kirkpatrick et al.). U.S. 4,847,371 (issued in July 1989 to Schara et al.) discloses a dual-atomization process and apparatus similar to those of the Pitchon et al. patents.

U.S. 4,465,702 (issued August 14, 1984 to Eastman) discloses a process for preparing a cold-water-swelling granular corn starch. An ungelatinized starch at 10-25 parts by weight is slurried in an aqueous C<sub>2</sub>-C<sub>3</sub> alkanol, 50-75 parts by weight, and about 13-20 parts water (provided that the alkanol and water mixture contains about 15-35 weight percent water including the water in the starch). The starch slurry is heated in a confined zone to a temperature of about 300-360°F for

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about 1 to about 30 minutes. The pregelatinized granular starch is then recovered from the slurry.

U.S. 5,037,922 (issued August 6, 1991 to Rajagopalan et al.) discloses a process for preparing cold-water-soluble granular starches. The starch granules are slurried in water and a polyhydric alcohol, such as, 1,3-propanediol, butanediol, or glycerol. The slurry is heated to 80-130°C for about 3-30 minutes to convert the crystalline structure of the granules to single helix crystals or to an amorphous state while maintaining the granular integrity of the starch. The starch is recovered from the liquid phase.

U.S. 5,149,799 (issued September 22, 1992 to Rubens) discloses a single atomization spray-drying process and an apparatus for preparing granular pregelatinized starches. The starch is slurried in an aqueous medium and a stream of the starch slurry is fed into an atomizing chamber within a spray nozzle at a pressure from about 50 to 200 psig. Steam is forced into the atomizing chamber at a pressure of 50 to 250 psig, and the starch is then simultaneously cooked and atomized as the steam forces the starch through a vent at the bottom of the chamber. The process takes place in a two-fluid, internal mix, spray nozzle. The process and the apparatus supply sufficient heat and moisture to the starch as it is being atomized to gelatinize the starch uniformly. The atomized starch is dried with a minimum of heat or shear effects as it exits the atomization chamber.

The thermally-inhibited granular starches and flours, both non-pregelatinized and pregelatinized, may be modified, e.g., by heat-, acid- and/or enzyme conversion, oxidation, phosphorylation, etherification, esterification, and/or chemical crosslinking. Usually these modifications are performed before the starch is

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thermally inhibited, but they may be performed after. These modifications are not preferred in applications where a food manufacturer requires a non-chemically modified starch.

5 The resulting thermally-inhibited starches and flours, both non-pregelatinized and pregelatinized, may be screened to select a desirable particle size or slurried in water or solvent, washed, filtered, and dried, bleached, or otherwise refined and modified. Also 10 the pH of the resulting starch may be adjusted.

Food products wherein the thermally-inhibited and pregelatinized starches are useful include thermally-processed foods, high acid foods, low acid food, dry mixes, refrigerated foods, frozen foods, extruded foods, 15 oven-prepared foods, stove top-cooked foods, microwaveable foods, full-fat or fat-reduced foods, and foods having low water activity. Food products wherein the thermally-inhibited starches are particularly useful are foods requiring a thermal processing step such as 20 pasteurization, retorting, or ultra high temperature (UHT) processing, as described in Volume 217 "Physical Principles of Food Preservation" by M. Karel et al., pp. 31-92 (Marcel Dekker, Inc., New York 1975).

The thermally-inhibited starches are 25 particularly useful in food applications where stability is required through all processing temperatures including cooling, freezing and heating. In food products subjected to temperature cycling operations, such as freeze-thaw cycling, a low temperature freeze-thaw stable 30 starch or flour, (e.g., waxy maize, waxy barley, waxy rice starch or flour or amaranth starch or flour, or the "V.O." hybrid waxy maize starch of U.S. 4,428,972) or a derivatized starch or flour (e.g., derivatized with hydroxypropyl groups) is preferred.

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The thermally-inhibited starches are also useful in food products where a non-chemically crosslinked starch thickener, viscosifier, gelling agent, or extender is required. Based on processed food 5 formulations, the practitioner may readily select the amount and type of thermally-inhibited or thermally-inhibited and pregelatinized starch or flour required to provide the necessary thickness and gelling viscosity in the finished food product, as well as the desired 10 texture. Typically, the starch is used in an amount of 0.1-35%, most preferably 2-6%, by weight, of the food product.

Among the food products which may be improved by the use of the thermally-inhibited starches or 15 thermally-inhibited and pregelatinized starches are high acid foods ( $\text{pH} < 3.7$ ) such as fruit-based pie fillings and baby foods, and the like; acid foods ( $\text{pH} 3.7-4.5$ ) such as tomato-based products; low acid foods ( $\text{pH} > 4.5$ ) such as gravies, sauces, and soups; stove top-cooked 20 foods such as sauces, gravies, and puddings; instant foods such as puddings; pourable and spoonable salad dressings; refrigerated foods such as dairy or imitation dairy products (e.g., yogurt, sour cream, and cheese); frozen foods such as frozen desserts and dinners; 25 microwaveable foods such as frozen dinners; liquid products such as diet products and hospital foods; and dry mixes for preparing baked goods, gravies, sauces, puddings, baby foods, hot cereals, and the like or dry mixes for predustring foods prior to batter cooking and 30 frying. The thermally-inhibited starches are also useful in preparing food ingredients such as encapsulated flavors and clouds.

Protein removal prior to thermal inhibition improves the flavor of the resultant thermally-inhibited 35 starches. A sodium chlorite extraction of the protein is

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exemplified hereafter. Other procedures which can be used for protein removal include washing the starch at an alkaline pH (e.g., pH 11-12) and/or treating the starch with proteases.

5

#### Sample Preparation

All the starches and flours used were granular and were provided by National Starch and Chemical Company of Bridgewater, New Jersey.

10

The controls for the test samples were from the same native sources as the test samples, were unmodified or modified as the test samples, and were at the same pH, unless otherwise indicated. Control samples were not dehydrated further or heat treated.

15

All starches and flours, both test and control samples, were prepared and tested individually.

20

The pH of the samples was raised by slurring the starch or flour in water at 30-40% solids and adding a sufficient amount of a 5% sodium carbonate solution until the desired pH was reached.

Measurements of pH, either on samples before or after the thermal inhibition steps, were made on samples consisting of one part starch or flour to four parts water.

25

The slurries were pregelatinized in a pilot spray dryer, Type 1-KA#4F, from APV Crepaco, Inc., Dryer Division, Attleboro Falls, Massachusetts, using a spray nozzle, Type 1/2J, from Spraying Systems Company of Wheaton, Ill. The spray nozzle had the following configurations: fluid cap 251376, air cap 4691312. The low initial cold viscosity samples were sprayed at a steam:starch ratio of 3.5-4.5:1, and the high initial cold viscosity samples were sprayed at a steam:starch ratio of 5.5-6.5:1.

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Moisture content of all samples after spray drying and before the dehydration step in the thermal inhibition process was 4-10%.

Except where a conventional oven or dextrinizer is used, the test samples were dehydrated and heat treated in a fluidized bed reactor, model number FDR-100, manufactured by Procedyne Corporation of New Brunswick, New Jersey. The cross-sectional area of the fluidized bed reactor was 0.05 sq meter. The starting bed height was 0.3-0.8 meter, but usually 0.77 meter. The fluidizing gas was air except where otherwise indicated and was used at a velocity of 15-21 meter/min. The side walls of the reactor were heated with hot oil, and the fluidizing gas was heated with an electric heater. The samples were loaded into the reactor and then the fluidizing gas introduced, or were loaded while the fluidizing gas was being introduced. No difference was noted in the samples in the order of loading. Unless otherwise specified, the samples were brought from ambient temperature up to 125°C until the samples became anhydrous or substantially anhydrous, and were further heated to the specified heat treating temperatures. When the heating temperature was 160°C, the time to reach that temperature was less than three hours.

The moisture levels of the samples at the final heating was 0%, except where otherwise stated. Portions of the samples were removed and tested for inhibition at the temperatures and times indicated in the tables.

These samples were tested for inhibition using the following Brabender Procedure.

Brabender Procedure

The pregelatinized thermally-inhibited granular starch to be tested was slurried in a sufficient amount of distilled water to give a 4.6% anhydrous solids starch

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slurry at pH 3 as follows: 132.75 g sucrose, 26.55 g starch, 10.8 g acetic acid, and 405.9 g water were mixed for three minutes in a standard home Mixmaster at setting #1. The slurry was then introduced to the sample cup of 5 a Brabender VISCO/Amylo/GRAPH fitted with a 350 cm/gram cartridge and the viscosity measured as the slurry was heated to 30°C and held for 10 minutes. The viscosity at 30°C and 10 minutes after hold at 30°C were recorded. The viscosity data at these temperatures are a 10 measurement of the extent of pregelatinization. The higher the viscosity at 30°C, the greater the extent of granular swelling and hydration during the pregelatinization process.

Heating was continued to 95°C and held at that 15 temperature for 10 minutes.

The peak viscosity and viscosity 10 minutes after 95°C were recorded in Brabender Units (BU) and used to calculate the percentage breakdown according to the formula:

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$$\% \text{ Breakdown} = \frac{\text{peak} - (95^\circ\text{C} + 10')}{\text{peak}} \times 100$$

25

where "peak" is the peak viscosity in Brabender Units and "(95°C + 10')" is the viscosity in Brabender Units at 10 minutes after 95°C.

If no peak viscosity was reached, that is, the 30 data indicated a rising curve or a flat curve, the viscosity at 95°C and the viscosity at 10 minutes after attaining 95°C were recorded.

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#### Characterization of Inhibition by Brabender Curves

As discussed above, characterization of a thermally-inhibited starch is made more conclusively by reference to a measurement of its viscosity after it is

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dispersed in water and gelatinized using the instrument described above.

For pregelatinized starches, the level of viscosity when dispersed in cold water will be dependent 5 on the extent to which the starch was initially cooked out during the pregelatinization process. If the granules are not fully swollen and hydrated during pregelatinization, gelatinization will continue when the starch is dispersed in water and heated. Inhibition is 10 determined by a measurement of the starch viscosity when the starch is dispersed at 4.6% solids in water at pH 3 and heated to 95°C.

When the pregelatinized starch has a high initial cold viscosity, meaning it was highly cooked out 15 in the pregelatinization process, the resulting Brabender traces will be as follows: for a highly inhibited starch, the trace will be a flat curve, indicating that the starch is already very swollen and is so inhibited starch it is resisting any further gelatinization, or the trace 20 will be a rising curve, indicating that further gelatinization is occurring at a slow rate and to a limited extent; for a less inhibited starch, the trace will show a dropping curve, indicating that some of the granules are fragmenting, but the overall breakdown in 25 viscosity will be lower than that for a non-inhibited control, or will show a second peak but the breakdown in viscosity will be lower than that for a non-inhibited control.

When the pregelatinized starch has a low 30 initial cold viscosity, meaning it was not highly cooked out in the pregelatinization process and more cooking is needed to reach the initial peak viscosity, the resulting Brabender traces will be as follows: for a highly inhibited starch, the trace will be a rising curve, indicating that further gelatinization is occurring at a 35

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slow rate and to a limited extent; for a less inhibited starch, the trace will show a peak viscosity as gelatinization occurs, and then a drop in viscosity, but with a lower percentage breakdown in viscosity than a control.

5

#### Brookfield Viscometer Procedure

Test samples are measured using a Model RVT Brookfield Viscometer and the appropriate spindle (the spindle is selected based on the anticipated viscosity of the material). In the applications described herein, spindles #3, #4 and #5 are typically used. The test sample, usually a cooked starch paste or food product, is placed in position and the spindle is lowered into the sample to the appropriate height. The viscometer is turned on and the spindle is rotated at a constant speed (e.g., 10 or 20 rpm) for at least 3 revolutions before a reading is taken. Using the appropriate conversion factors, the viscosity (in centipoises) of the sample is recorded.

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#### Bostwick Consistometer Procedure

With the sample cup in the closed position, the test material is loaded into the cup and leveled-off. The gate is opened and the test material is allowed to flow. The distance traveled (in centimeters) over a specific time period (e.g., 60 seconds) is recorded.

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#### Examples

The following examples will more fully illustrate the embodiments of this invention. In the examples, all parts and percentages are given by weight, all temperatures are in degrees Celsius unless otherwise noted. The resultant thermally-inhibited starches are referred to as "T-I" starches unless otherwise indicated.

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The conditions used for the pH adjustment, if any, and heat treatment are indicated. The time is the heating time at that temperature. When a zero hold time is indicated (e.g., 160°C/0 min), it means the sample was taken as soon as the material reached that particular temperature (e.g., 160°C). The initial pH of the starch prior to the dehydration step is indicated. Where the pH of the starch is adjusted, the pH adjustments are done with sodium carbonate unless specified otherwise.

10

EXAMPLE 1

This example illustrates the preparation of pregelatinized and thermally-inhibited granular starches wherein the pregelatinization step, as described below, is carried out prior to the thermal inhibition process. The fluidized bed process described previously was used.

Starch slurries (30-40% solids), pH adjusted to 6, 8, and 10, with a 5% sodium carbonate solution were pregelatinized in a pilot size spray drier, Type-1-KA#4F, from APV Crepaco, Inc., Dryer Division, of Attleboro Falls, Massachusetts, using a spray nozzle, Type 1/2 J, from Spraying Systems Company of Wheaton, Illinois. The spray nozzle had the following configuration: fluid cap, 251376, and air cap, 4691312.

The resultant pregelatinized thermally-inhibited granular starches were evaluated for inhibition. The results are shown below:

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	High Initial Viscosity Pregel Waxy Maize (pH 6.0)	Viscosity (B.U.)				Break- down (± 2) (%)
		30°C	30°C + 10 min	Peak	95°C	95°C + 10 min
5	Control	1280	960	--	170	90
10	Time at 160°C (min)					--
	0	700	980	700	610	370
	30	600	910	720	690	370
15	90	450	780	915	740	400
	150	360	590	925	800	500
20	Low Initial Viscosity Pregel Waxy Maize (pH 6.0)	Viscosity (B.U.)				Break- down (± 2) (%)
25	Control	230	250	750	340	100
30	Time at 160°C (min)	.				87
	30	100	130	600	370	210
	60	100	140	730	500	260
	120	100	130	630	430	260
35	180	90	120	550	390	240
						56

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High  
Initial  
Viscosity  
Pregel  
Waxy  
Maize  
(pH 8.0)

Break-  
down  
(± 2)  
(%)

		Viscosity					Break-down (± 2) (%)
		30°C	30°C + 10 min	Peak	95°C	95°C + 10 min	
	Control	1400	1020	--	270	100	--
10	Time at 160°C (min)						
	0	700	1060	1050	760	280	73
	60	260	600	1340	1200	780	42
15	90	240	440	1280	1240	1000	22
	120	280	420	1320	1320	1280	3
	150	120	200	860	860	820	7
	180	180	260	980	980	920	8

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Low  
Initial  
Viscosity  
Pregel  
Waxy  
Maize  
(pH 8.0)

Break-  
down  
(± 2)  
(%)

		Viscosity					Break-down (± 2) (%)
		30°C	30°C + 10 min	Peak	95°C	95°C + 10 min	
30	Control	250	250	820	340	130	84
	Time at 160°C (min)						
	0	50	100	690	460	270	61
35	60	40	50	840	590	320	62
	120	20	30	720	650	450	38
	180	20	30	590	570	450	24

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	High Initial Viscosity Pregel Waxy Maize (pH 10)	viscosity (B.U.)				Break- down (± 2) (#)
		<u>30°C</u>	<u>30°C + 10 min</u>	<u>Peak</u>	<u>95°C</u>	<u>95°C + 10 min</u>
5	Control	1010	740	--	300	160
10	Time (min)					
	140°C/0	550	850	1280	1080	750
	150°C/0	270	420	1680	1680	1540
	160°C/0	170	240	--	1180	1440
15	160°C/30	80	85	--	410	650
	160°C/60	60	60	--	150	300
	160°C/90	50	50	--	80	140
	160°C/120	40	40	--	80	130
	160°C/150	40	40	--	60	90
20	160°C/180	40	40	--	45	70

<sup>1</sup> ris.visc. is equal to rising viscosity.

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	Low Initial Viscosity Pregel Waxy Maize (pH 10)	<u>Viscosity</u> (B.U.)				Break- down (± 2) (%)
		<u>30°C</u>	<u>30°C + 10 min</u>	<u>Peak</u>	<u>95°C</u>	<u>95°C + 10 min</u>
5	Control	200	190	615	350	190
10	Time (min)					--
	130°C/0	110	180	1500	880	530
	150°C/0	50	80	1670	1540	1250
	160°C/0	30	30	--	1040	1320
15	160°C/30	30	30	--	380	640
	160°C/60	30	30	--	150	310
	160°C/90	10	10	--	50	120

1 ris.visc. is equal to rising viscosity.

20           The data show some thermal inhibition was attained in all cases and that increasing the initial pH and the time of heating increased the level of inhibition. For the samples at pH 6.0, at 0 and 30 minutes, the recorded peak was actually a second peak obtained after the initial high viscosity began to breakdown. For some of the samples at pH 10, no peak viscosity was reached, indicating a highly inhibited starch.

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EXAMPLE 2

This example shows that the thermally-inhibited starches and flours are essentially sterilized by the heat-treatment and remain sterile when properly stored.

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Part A - Thermally-Inhibited Waxy Rice Flour

The flour was adjusted to pH 9.5 and thermally-inhibited in the fluidized bed as previously described and stored for about 3 months in a non-sterilized, covered glass container. The thermally-inhibited flours and the control flour were microbiologically tested for their total plate count using the procedure described on pages 17-19 of Chapter 3 "Aerobic Plate Count" by J.T. Peeler and L.J. Maturin, FDA Bacteriological Analytical Manual, 7th Ed. (A.O.A.C. International, Arlington, Va. 1992). The results are shown below:

		<u>Plate Count</u> (CFU) <sup>1</sup>
20	Waxy Rice Flour (no heat treatment)	7500
25	T-I Waxy Rice Flour (160°C/0 min sample)	<10
30	T-I Waxy Rice Flour (160°C/60 min sample)	<10
	T-I Waxy Rice Flour (160°C/120 min sample)	<10

<sup>1</sup> Colony forming units.

35

Part B - Thermally-Inhibited Waxy Maize Starch

The starch was adjusted to pH 9.5 and thermally-inhibited in the fluidized bed as previously described and stored for about 2 months in non-sterilized, covered glass containers. The thermally-inhibited starches and the control starch were

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microbiologically tested for their total plate count using the above procedure. The results are shown below.

		<u>Plate Count</u> (CFU) <sup>1</sup>
5	Waxy Maize Starch (no heat treatment)	2000
10	T-I Waxy Maize Starch (160°C/0 min sample)	<10
	T-I Waxy Maize Starch (160°C/60 min sample)	<10
15	T-I Waxy Maize Starch (160°C/120 min sample)	<10
20	<sup>1</sup> Colony forming units.	

The above results are particularly interesting, especially since the thermally-inhibited flours and starches were not handled using aseptic techniques. If stored and maintained under sterile conditions, these starches should be useful in products where microbiological considerations are of concern.

30

EXAMPLE 3

This example shows the effect of protein removal on the flavor (i.e., taste and smell) of a thermally-inhibited waxy maize.

Prior to the thermal inhibition process, the protein was extracted from a waxy maize starch as follows. The starch was slurried at W=1.5 (50 lbs starch to 75 lbs of water) and the pH was adjusted to 3-3.5 with sulfuric acid. Sodium chlorite was added to give 2% on the weight of the starch. The starch was steeped overnight at room temperature. The pH was raised to about 9.5 using a 3% sodium hydroxide solution and washed well prior to drying. The protein level of the starch

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was reduced to about 0.1%. The protein extracted starch and untreated starch were heat-treated in the fluidized bed as previously described. The protein level of the thermally-inhibited waxy maize control (pH 9.5) was about 5 0.3%.

Using a one-sided, directional difference taste testing procedure, as described in "Sensory Evaluation Techniques" by M. Meilgaard et al., pp. 47-111 (CRC Press Inc., Boca Raton, Florida 1987), the protein-reduced 10 thermally-inhibited waxy maize (pH 9.5; 160°C/90 min) was compared to the thermally-inhibited waxy maize (pH 9.5; 160°C/90 min) which had not been protein-reduced prior to heat treatment.

For the taste test, 3% starch cooks (samples 15 heated at 100°C for 15 min) were prepared and panelists were asked to select which sample was "cleaner" in flavor. All tests were done in a sensory evaluation room under red lights in order to negate any color differences that may have been present between samples. The results 20 are shown below:

<u>Trial #</u>	<u>Number of Panelists</u>	<u>Number of Positive Responses<sup>1</sup></u>	<u>Significance Level (<math>\alpha</math> risk)<sup>2</sup></u>
1	15	12	5%
25	2	14	5%

1 The number indicates those respondents who selected the protein-reduced product as being cleaner in flavor.

30 2 The  $\alpha$  values were determined from a statistical table. An  $\alpha$  risk of 5% indicates (with 95% confidence) that the samples are statistically different, i.e., that the protein-reduced product is cleaner than the control.

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The results show that protein removal prior to the heat treatment helps to improve the flavor of the thermally-inhibited waxy maize starches.

5

EXAMPLE 4

This example shows the use of pregelatinized thermally-inhibited granular waxy maize starches in an instant chocolate pudding. All of starches were adjusted to pH 10 and pregelatinized using the procedure described earlier for preparing granular pregelatinized starches.

10 The ingredients are set out below.

	<u>Ingredients</u>	<u>%</u>		
		#1	#2	#3
15	Sugar	51.22	51.22	51.22
	Dextrose	18.00	18.00	18.00
	Pregelatinized Granular Waxy Maize (pH 10)	15.30	--	--
	T-I	--	15.30	--
20	Pregelatinized Granular Waxy Maize (pH 10; 140°C/0 min)			
	T-I	--	--	15.30
	Pregelatinized Granular Waxy Maize (pH 10; 160°C/0 min)			
25	Cocoa (Dezaan)	7.50	7.50	7.50
	Tetrasodium Pyrophosphate	2.40	2.40	2.40
	Caramel Color	2.00	2.00	2.00
	Disodium Phosphate	1.20	1.20	1.20
30	Vegetable Oil	0.80	0.80	0.80
	Myvacet 9-45	0.50	0.50	0.50
	Salt	0.50	0.50	0.50
	Chocolate Flavor	0.30	0.30	0.30
35	Vanilla Flavor	0.20	0.20	0.20
	Brown Color	0.08	0.08	0.08
	Total	100.00	100.00	100.00

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The Myvacet 9-45 and the vegetable oil were preblended with a portion of the sugar. All the remaining ingredients were thoroughly dry blended and ground in a coffee grinder. One part of the dry mix was added to 3.51 parts of cold milk, mixed for 2 minutes on medium speed, and refrigerated to set.

5 The evaluations are shown below.

Instant Chocolate Pudding Evaluation

	<u>Texture</u>	<u>Taste</u>	<u>Brookfield Viscosity (cps)</u>
10	#1 smooth, soft, cohesive	quick meltdown, smooth,	20,000
	#2 short, cuttable, non-cohesive	quick meltdown, smooth,	16,000
	#3 short, cuttable, non-cohesive	quick melt, smooth,	13,200

15

The texture of the thermally-inhibited products was less gummy and had a smoother, creamier mouthful compared to the waxy maize control which possessed an undesirable cohesive texture.

20

EXAMPLE 5

This example illustrates the preparation of instant imitation grape jellies employing pregelatinized granular starches which are prepared as previously described and subsequently thermally-inhibited.

25 The ingredients are shown below:

30	Pregelatinized granular T-I Corn (pH 8.5; 150°C/ 0 min)	5.95%	---
	Pregelatinized granular T-I Potato (pH 9.5; 150°C/0 min)	---	5.95%

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	Sugar	29.00%	29.00%
	Sodium Benzoate	0.09%	0.09%
	Unsweetened Concord Grape Juice	45.00%	45.00%
5	Water	19.96%	19.96%
	Total	100.00%	100.00%

The solid ingredients are blended together and  
 10 added to the liquids in the mixing bowl of a Sunbeam  
 Mixmaster mixer and mixed on low speed for 2-3 minutes  
 The resulting mixture is refrigerated for a minimum of 4  
 hours.

15 The resultant jellies should have a clean-cutting jelly texture.

#### EXAMPLE 6

This example describes the preparation of a no-fat French salad dressing. The starch is pregelatinized 20 using the procedure previously described.

The ingredients are shown below:

	<u>Ingredient</u>	<u>%</u>
	Water	48.14
25	Sugar	11.51
	Tomato paste (25% solids)	6.5
	Salt	1.0
	Mustard Powder	0.3
	Onion Powder	0.3
30	Garlic Powder	0.2
	Keltrol F (gum)	0.35
	Keloid LVF (gum)	0.3
	MSG	0.5
	Oleoresin Paprika	0.12
35	Sodium Benzoate	0.08
	Waxy Maize	2.5

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<u>Ingredient</u>	<u>%</u>
50 Grain white vinegar	24.0
TiO <sub>2</sub>	0.15
Potassium sorbate	0.08
5 Pregelatinized T-I Waxy Maize (pH 9.5; 160°C/0 min)	4.08
Total	100.00

10 The French salad dressings are prepared by blending the dry ingredients, adding the blend to water in a Hobart Mixer, and mixing the aqueous dispersion for 10 minutes at #2 speed. The tomato paste, oleoresin, and paprika are blended, added to the aqueous dispersion, and  
15 mixed for 2 minutes at #2 speed. The oil is slowly added at #2 speed, followed by the vinegar. The mixture is mixed for one minute at #3 speed and then put through a Charlotte colloid mill set at a clearance of 0.03 inches. The resultant dressing should have a smooth and creamy  
20 texture.

#### EXAMPLE 7

25 This example describes the preparation of a flavored particle for inclusion in a muffin mix which is prepared using a thermally-inhibited pregelatinized corn starch which is pregelatinized using the procedure previously described.

30 The following dry ingredients are mixed to a homogeneous blend in a ribbon blender:

Sweet whey: 17.2 parts

Dextrose (as corn syrup solids): 14.9 parts

35 Sucrose (95%): 7.4 parts

Wheat flour: 31.2 parts

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Pregelatinized T-I Corn Starch 6.5 parts  
(pH 9.0; 160°C/15 min)

5

The following ingredients are mixed to a homogeneous dispersion of solids and liquids:

10 Liquid corn oil: 13.7 parts

Glycerine: 7.7 parts

USDA blue color: 1.1 parts

15 Imitation blueberry flavor: 0.2 part

Citric acid: 0.1 part

20 The dispersion is slowly added to the dry ingredients in the ribbon blender and mixing at room temperature is continued until a homogeneous semi-solid dispersion is obtained. Sufficient water is added during mixing to provide a moisture content of 15%.

25 The semi-solid dispersion is passed through a California Pellet mill and cut into particles of approximately cylindrical shape having a diameter of about  $\frac{1}{8}$  inch and a length of about  $\frac{1}{2}$  inch. The particles are semi-soft in the nature of berry pulp. They can be 30 stored at room temperature for at least 1 year without significant deterioration of the semi-soft and semi-moist texture or loss of flavor.

Now that the preferred embodiments of the present invention have been described in detail, various 35 modifications and improvements thereto will become readily apparent to those skilled in the art.

Accordingly, the spirit and scope of the invention are to be limited only by the appended claims and foregoing specification.

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WHAT IS CLAIMED IS:

1. A food which comprises, as a food ingredient, a thermally-inhibited pregelatinized granular starch which is pregelatinized prior to or after being thermally-inhibited.

2. A food which comprises, as a food ingredient, a thermally-inhibited pregelatinized granular flour which is pregelatinized prior to or after being thermally-inhibited.

3. The food of Claim 1, wherein the food is a thermally processed food.

4. The food of Claim 3, wherein the thermally processed food is a high acid food having a pH of less than about 3.7, an acid food having a pH of about 3.7-4.5, or a low acid food having a pH of greater than about 4.5.

5. The food of Claim 1, wherein the food is a dry mix for preparing cooked foods or for dusting foods to be battered and cooked, a refrigerated food, a frozen food, an extruded food, a stove top-cooked food, an oven-prepared food, a microwaveable food, or a full-fat, low-fat or no-fat food.

6. The food of Claim 5, wherein food is a frozen food and wherein the starch is a freeze-thaw stable, thermally-inhibited starch selected from the group consisting of waxy maize, V.O. waxy maize, waxy rice, waxy barley, and amaranth, or a thermally-inhibited waxy rice flour, or a thermally-inhibited derivatized starch.

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7. The food of Claim 1, wherein the food is a yogurt, a sour cream, a cheese cake, a sauce selected from the group consisting of a white sauce, cheese sauce, a barbecue sauce, and a tomato sauce, a fried food, an emulsified meat, an ice cream, a frozen dessert, a frozen dinner, a baby food, a pie filling, a gravy, or a bakery product.

8. The food of Claim 1, wherein the starch is a cereal, a root, a tuber, a legume, or a fruit starch.

9. The food of Claim 2, wherein the flour is a cereal, a root, a tuber, a legume, or a fruit flour.

10. The food of Claim 8, wherein the starch is selected from the group consisting of corn, pea, potato, sweet potato, barley, wheat, rice, sago, banana, amaranth, tapioca, sorghum, V.O. hybrid waxy maize, waxy maize, waxy rice, waxy barley, waxy potato, a starch containing greater than 40% amylose, and combinations thereof.

11. The food of Claim 9, wherein the starch is selected from the group consisting of corn, pea, potato, sweet potato, barley, wheat, rice, sago, banana, amaranth, tapioca, sorghum, V.O. hybrid waxy maize, waxy maize, waxy rice, waxy barley, waxy potato, a starch containing greater than 40% amylose, and combinations thereof.

12. The food of Claim 1, wherein the starch is waxy starch selected from the group consisting of waxy maize, waxy rice, waxy potato, and waxy barley.

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13. The food of Claim 1, wherein the starch is a gelling starch selected from the group consisting of corn, high amylose corn, wheat, and rice starch.

14. The food of Claim 1, wherein the food is a dry mix for preparing instant foods or cooked foods or for dusting foods to be battered and breaded; a refrigerated food; a frozen food; an extruded food; a thermally-processed food selected from the group consisting of a high acid food having a pH of less than about 3.7, an acid food having a pH between about 3.7 and 4.5, and a low acid food having a pH of about 4.5 or greater; a stove top-cooked food; a microwaveable food; or a full-fat, low-fat, or no-fat food.

15. The food of Claim 2, wherein the food is a dry mix for preparing instant foods or cooked foods or for dusting foods to be battered and breaded; a refrigerated food; a frozen food; an extruded food; a thermally processed food selected from the group consisting of high acid food having a pH of less than about 3.7, an acid food having a pH between about 3.7 and 4.5, and a low acid food having a pH of about 4.5 or greater; a stove top-cooked food; a microwaveable food; or a full-fat, low-fat, or no-fat food.

16. The food of Claim 3 wherein the food is a baked good; a baby food; a cereal; a snack; a sauce; a gravy; or a pasta.

17. The food of Claim 4 wherein the food is a baked good; a baby food; a cereal; a snack; a sauce; a gravy; or a pasta.

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18. A method for preparing a food which is to be extended, thickened or gelled without heating, which comprises the step of adding to one or more food ingredients, a thermally-inhibited and pregelatinized granular starch or thermally-inhibited and pregelatinized flour which is pregelatinized prior to or after being thermally-inhibited.

## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 95/00682

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 A23L1/0522

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 A23L A21D C08B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US,A,4 391 836 (CHIU CHUNG-WAI) 5 July 1983 see examples VIII-X	1-18
A	GB,A,1 479 515 (GERBER PRODUCTS COMPANY) 13 July 1977	---
A	US,A,2 373 016 (RAYMOND E. DALY ET AL.) 3 April 1945	---
A	US,A,3 977 897 (WURZBURG ET AL.) 31 August 1976	-----

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

## \* Special categories of cited documents:

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
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- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

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\*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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## INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No  
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Patent document cited in search report	Publication date	Patent family member(s)		Publication date
US-A-4391836	05-07-83	-----	NONE	-----
GB-A-1479515	13-07-77	-----	NONE	-----
US-A-2373016	03-04-45	-----	NONE	-----
US-A-3977897	31-08-76	FR-A, B GB-A- JP-C- JP-A- JP-B- NL-A-	2322925 1505039 1050860 52038037 55042640 7609906	01-04-77 22-03-78 26-06-81 24-03-77 31-10-80 10-03-77

Form PCT/ISA/210 (Patent family members) (July 1992)